Laboratory Annealing of Glass

1 Scope

1.1 Introduction

This document provides for the laboratory annealing of glass particles by Geologist-Forensic Examiners within the Trace Evidence Unit (TEU). In glass manufacturing, annealing is the process of reducing residual strain in glass by controlled heating and cooling. The technique described in this document applies to flat glass used for windows, doors, display cases, and mirrors; automotive glass; optical glass; and glass objects that have been in a fire.

The thermal history of glass fragments is a useful property in determining glass source classifications. When questioned glass is not distinguishable in refractive index (RI) from the known glass, both may be annealed in the Laboratory to determine whether or not they have similar thermal histories. Because annealing alters the optical properties of a glass fragment, in most cases, RI should be measured before laboratory annealing. After the sample is annealed in the Laboratory, its RI can be measured again. RI generally increases after laboratory annealing and the magnitude of change in RI (ΔRI) may be used to classify fragments as originating from tempered or non-tempered sources.

Laboratory annealing is also useful in the determination and comparison of refractive indices of glass samples that have been subjected to fire (e.g., arson cases). A glass object that has been heated in a fire may be thermally altered, and therefore the RI of the glass would be changed. This change is unpredictable and is not typically uniform across a glass object. For such samples, laboratory annealing before measuring refractive indices will remove strain introduced as a result of the fire so that a meaningful comparison may be performed.

1.2 Definitions

1.2.1 The *annealing point* is the temperature at which the strain in glass will be relieved. See Table 1 for the approximate annealing points of glass types commonly seen in casework.

1.2.2 The *annealing range* spans the upper limit of the annealing region, slightly above the annealing point, through the strain point.

1.2.3 Stresses engendered in a piece of glass by heating and cooling may be *temporar*y until the glass cools to a lower temperature at which it acquires *ordinary permanent strain*.

1.2.4 The *softening point* is the minimum viscosity that the worked mass of glass must have before being allowed to stand on its own (or it will deform under its own weight).

1.2.5 The *strain point,* or the lower limit of the annealing region, is the point below which no additional permanent strain will be introduced in the glass. The strain remaining in the glass is directly proportional to the cooling rate prior to reaching the *strain point*.

Table 1. Common Glass Types and Their Approximate Viscosity Data

Glass Code	Type	Strain Point ºC	Annealing Point ºC	Softening Point ºC
0080	Soda Lime (Tubing)	395	435	630
0281	Soda Lime (General Purpose)	490	530	707
7250	Borosilicate (Baking Ware)	485	530	780
7740	Borosilicate (General Purpose)	520	565	820

Corning Glass Works, Corning, NY, 1957

2 Equipment/Materials/Regents

- Ceramic spot plate or equivalent
- Forceps
- Insulated gloves
- Laboratory coat
- Programmable muffle furnace or equivalent
- Safety goggles
- Additional materials may be used at the discretion of the Geologist-Forensic Examiner.

3 Standards and Controls

Not applicable.

4 Sampling or Sample Selection

4.1 The procedures for the collection and processing of glass from submitted items of evidence can be reviewed in the TEU Evidence Processing Procedures.

4.2 Glass fragments selected to be annealed should be large enough to split in two pieces: one to determine the pre-annealed RI value and one to determine the post-annealed RI value. Smaller fragments may be annealed at the discretion of the Geologist-Forensic Examiner.

5 Procedure

5.1 Place each glass fragment to be annealed into an individual well in a ceramic spot plate(s) or equivalent.

5.2 Note the location of each fragment. Labeling the spot plates with a "permanent" marker is not a sufficient labeling method as the ink will ash at high temperatures.

5.3 Load the samples into the muffle furnace. When space allows, all fragments to be compared will be annealed simultaneously.

5.4 Close the door of the muffle furnace.

5.5 Place the power switch to the ON position.

5.6 Program the muffle furnace according to the following conditions:

5.6.1 Ramp the temperature in the furnace up to the set point at a rate no greater than 25ºC per minute. The set point should be chosen to be above the *strain point* and below the *annealing point* typical for glass of the type being annealed (see Table 1). 525ºC is sufficient for most glasses commonly encountered in forensic casework.

5.6.2 Hold the temperature at the set point for at least 30 minutes.

5.6.3 Set the cooling rate to slowly cool the glass at approximately 1ºC/minute through the suspected *strain point* (see Table 1). Other cooling rates may be programmed at the discretion of the Geologist-Forensic Examiner. When the *annealing point* is not known with any great accuracy, the slow cooling should continue to 375°C, which is below the *strain point* for most glasses commonly encountered in casework.

5.6.4 Below the suspected *strain point*, the cooling rate can be set at a faster rate at the discretion of the Geologist-Forensic Examiner, up to 5ºC/minute. Alternately, the muffle furnace may be turned off and allowed to cool undisturbed with the door closed. Do not open the door of the muffle furnace until the temperature control reads less than 200ºC.

5.7 Run the program.

6 Calculations

Not applicable.

7 Measurement Uncertainty

Not applicable.

8 Limitations

8.1 Annealing changes the RI of sample.

8.2 Some glass fragments may be too small to recover without the risk of losing them.

8.3 The use of the other annealing protocols such as the Locke "short schedule" can result in lower ΔRI values than those obtained using longer annealing schedules.

8.4 Upon annealing, heat-strengthened glass has a ΔRI intermediate between that of non-tempered and tempered glass. Therefore, it is not always possible to classify glass type using ΔRI alone.

8.5 Extremely small glass fragments can be lost during annealing.

8.6 Glass with an unusual composition may have a higher *strain point* and *annealing point* than the glasses listed in Table 1. Glasses with unusual compositions may not be fully annealed even when heated to 525°C.

9 Safety

9.1 Appropriate eye protection and gloves will be used when handling broken glass.

9.2 Eye protection and gloves will be used when breaking glass.

9.3 Always wear insulated gloves when handling hot materials.

10 References

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Rev. #	Issue Date	History
1	02/07/2018	Updated throughout removing references to TEU where appropriate. Updated Section 1 Scope to include Geologist/Forensic Examiner and throughout document when appropriate. Deleted Section 4 Calibration and document renumbered. Updated new Section 4 to add 'Sample Selection' and minor edits. 'Specimen' changed to 'sample' throughout document. Section 5.3: "Q and K" deleted. Updated Sections 9.1 and 9.2 to change 'should' to 'will'. References updated in Section 10 and throughout document.
2	09/01/2021	Added "Additional materials..." to the Section 2 Equipment list. Changed "Mineralogy" to "Geology in Approval Section. Updated TL in Approval Section.

Approval Redact - Signatures on File

Trace Evidence Unit Chief _____ Date: 08/31/2021

Geology Technical Leader _____ Date: 08/31/2021